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Observations and issues on mechanisms of grain refinement during ECAP process

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Abstract

The equal channel angular pressing route, defined by rotating schemes between adjacent passes, significantly affects effectiveness of grain refinement. It is of interest to study the mechanisms of grain refinement. Previous work has considered the accumulative strain and the effects of shear strain plane in the interpretation of certain experimental observations. However, they are not sufficiently general, and contradict each other in some cases. In this paper, we analyze experimental results available in the literature, and investigate the fundamental mechanisms of grain refinement. We believe that the interaction of shear plane with texture and crystal structure plays a primary role in grain refinement, while the accumulative strain plays a secondary role. Our model can explain the experimental results in the literature very well. Issues on the grain refinement are discussed and further research to solve these issues is suggested. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Grain refinement; Equal channel angular pressing; Mechanisms

1. Introduction

A major challenge to producing ultrafine-grained (UFG) materials (grain size in the range 10–1000 nm) for many structural applications is the difficulty in fabricating them into bulk form. Equal-channel angular pressing (ECAP) is at present one of the most promising techniques that can process bulk UFG materials large enough for structural applications [1–3]. Briefly, in this technique, a metal billet is pressed through a die containing two channels, equal in cross-section, intersecting at an angle Φ. During the pressing, the billet undergoes severe shear deformation but retains the same cross-sectional geometry so that it is possible to repeat the pressings for a number of passes, each one refining grain size. Between each adjacent two passes, it is possible to rotate the billet around its longitudinal axis, creating different ECAP routes [4–15]. The processing route significantly affects the grain refinement and grain shape. It is of interest to find an ECAP route that most efficiently refines the grain size.

Four rotation routes have been used to systematically study the microstructural development during the ECAP [4–14,16,17]. Fig. 1 illustrates the four ECAP routes: route A, the billet is not rotated; route B_C, the billet is rotated 90° clockwise; route B_A, the billet is rotated 90° clockwise and counterclockwise alternatively; route C, the billet is rotated 180°.

Another factor that significantly affects the microstructural development is the channel-intersection angle, Φ, which determines the shear strain from an individual pass [18]:

$$\gamma = 2\cot\left(\frac{\Phi}{2}\right) \tag{1}$$

Smaller Φ will result in higher shear strain from each pass and therefore will be more effective in refining grains [9]. Values of Φ ranging from 90 to 157.5° have been reported in the literature [6,9].

Materials that have been used for studying the effect of ECAP route on grain refinement and microstructural development include Cu [4,19], Al and Al alloys [4–9,12,13], Ni [14], iron and steel [20–22], and Ti and Ti alloy[23–25]. The most thorough studies were carried out on an Al alloy by Prangnell et al. [6], and on pure Al by Iwahashi et al. [7,13] and Oh-Ishi et al. [8].

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However, our understanding of the grain refinement during the ECAP is still very superficial, and contradicting conclusions have been reported [6,7,13]. It is the objective of this paper to analyze these observations and to relate them to deformation fundamentals such as shear plane, crystal structure, shear strain and deformation-induced texture. A hypothesis on the mechanisms of grain refinement will be presented. Issues concerning the hypothesis will be discussed and future research necessary to address these issues will be suggested.

2. Observations

Prangnell et al. [6] processed an Al–3% Mg–0.2% Zr–0.2% Fe alloy with all four ECAP routes using a die with a channel-intersection angle $\Phi = 120°$. High-resolution electron back-scattered diffraction was used to quantitatively measure grain orientation. Their results show that route A is most effective in refining grains and generating high-angle grain boundaries, while route C is the least effective. Route B_C is slightly more effective than route B_A. They argued that route A is most effective in refining grains because it does not produce redundant strain. When the billet is rotated 180° from a previous pass, the shear strain is reversed, resulting in redundant strain, which may reverse the microstructure and texture produced during the previous ECAP pass [4,26,27]. In route C, the shear strain of one pass is reversed by the strain from the following pass, making it least effective in refining the grain size. In route B_C, shear strains imposed by every other pass reverse each other. For example, the third pass reverses the strain from the first pass and the fourth pass reverses the strain from the second pass. This makes route B_C less effective than route A but more effective than route C. The redundant strain theory is consistent with observations of microstructures obtained by a face-centered cubic (f.c.c.) Al alloy with a $\Phi = 120°$ die. However, it cannot explain why route B_C is slightly more effective than route B_A, which does not have redundant strain.

Iwahashi et al. [7,13] processed pure Al with ECAP routes A, B_C and C using a die with $\Phi = 90°$. They concluded that route B_C is most effective and route A is least effective in grain refinement. Oh-Ishi et al. [8] compared routes B_C and B_A using a die with $\Phi = 90°$, and concluded that route B_C is more effective than route B_A in grain refinement. Langdon et al. [28] summarized the effectiveness of ECAP routes in grain refinement as route $B_C >$ route $C >$ routes A and B_A. To explain these experimental observations, Furukawa et al. [16] analyzed the distortion of a cubic element caused by deformation from each ECAP routes. They concluded that routes B_C and C are more effective than routes A and B_A, because the cubic element is restored after $4n$ passes in route B_C and $2n$ passes in route C, while routes A and B_A deform the cubic element continuously, where n is an integer. Such an explanation is superficial and does not address the fundamental mechanism of grain refinement. Later work by Iwahashi et al. [7,12] and Nemoto et al. [17] went a step further to consider the orientation relationships of shearing strain planes of consecutive passes from each ECAP route. However, they also failed to address the more fundamental issue of how and why these shearing planes affect the grain refinement. Not surprisingly, although these models can explain the experimental results of Al and Al alloys processed by ECAP dies with a Φ angles of 90°, they contradict the results of Prangnell et al. [6], where $\Phi = 120°$.

Nakashima et al. [9] studied the effect of channel-intersection angle Φ on the grain refinement by processing pure Al using dies with Φ angles of 90, 112.5, 135 and 157.5°. Route B_C was used for the study. They found that for the same amount of strain, the grain refinement is most effective with $\Phi = 90°$. They attributed the effective grain refinement of the 90° die to the 60° angle between the two shearing planes (see Fig. 2). However, the shearing plane rationale cannot explain why route B_A is less effective than the route B_C since adjacent planes in route B_A also are at a 60° angle. Another puzzle is why route C is more effective than route A when $\Phi = 90°$, which is contrary to the observations of Prangnell et al. where $\Phi = 120°$.

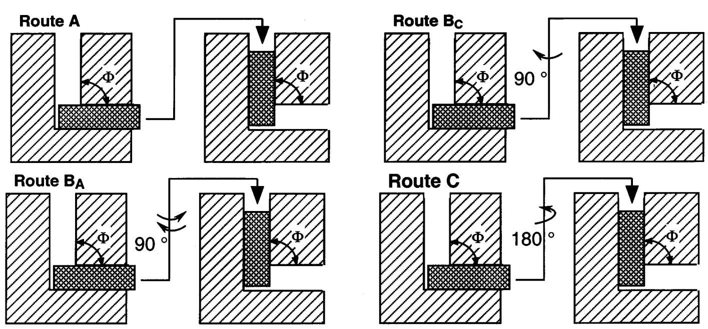

Fig. 1. Rotation schemes of four ECAP routes.

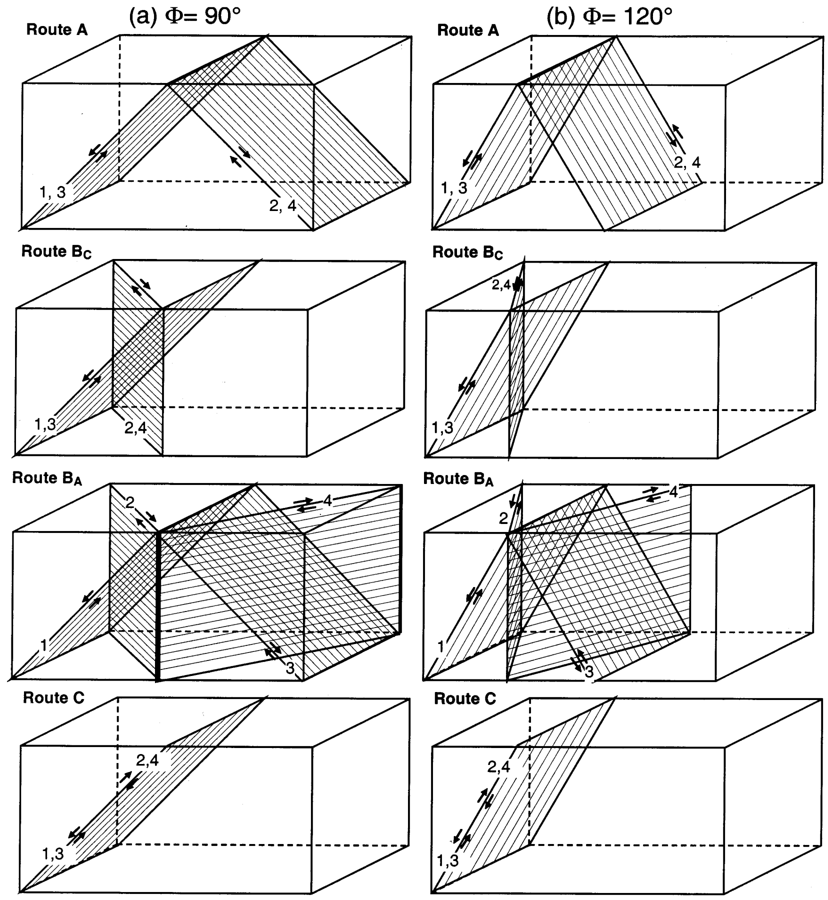

Fig. 2. Shear strain planes for each ECAP routes for dies with (a) $\Phi = 90°$ and (b) $\Phi = 120°$.

Segal [29] proposed that shear bands and subgrain rotation determines the grain refinement during the ECAP. However, his model is vague and needs further experimental validation. In addition, the same explanation that fits the die angle $\Phi = 90°$ cannot explain the results of Prangnell et al., where $\Phi = 120°$. Other researchers [17,21,22,30] have studied the development of microstructures, and generally observed that the misorientation between subgrains increases with increasing number of ECAP passes. Some of these subgrains will become grains with high-angle grain boundaries as the number of ECAP passes increases. These studies did not address the effectiveness of ECAP routes in grain refinement.

We believe that the explanation of relative effectiveness of grain refinement must consider both the crystal structure and mechanics of material flow during ECAP. Only the f.c.c. structure of Al and an Al alloy has been analyzed in the aforementioned studies. Sufficiently thorough experiments including detailed microstructural analysis have not yet been conducted for materials with other crystal structures. However, preliminary results for body-centered cubic (b.c.c.) and hexagonal close-packed (h.c.p.) metals have been reported. Segal [14] processed Ni, which has a b.c.c. structure, with routes A and C using a $\Phi = 90°$ die. Unfortunately, no quantitative comparison on their effectiveness in grain refinement was attempted. However, optical micrographs show that the angle between grain elongation direction and the extrusion axis becomes smaller with increasing number of passes for route A.

Stolyarov et al. [23] processed h.c.p. Ti with routes B_C, B_A and C using a $\Phi = 90°$ die. They found that route B_C is most effective in refining grain size and route B_A is least effective.

The apparent inconsistencies in these observations may be resolved by careful analysis and hypotheses, as proposed in the following sections.

3. Analysis

The process of grain refinement during large strain deformation has been discussed by Hughes et al. [31,32], Liu et al. [33], Sun et al. [34], Humphreys et al. [35] and Davenport et al. [27]. With increasing strain, cell blocks separated by geometrically necessary boundaries (GNBs) and cellular structures with incidental dislocation boundaries (IDBs) form within the grains. The misorientations of GNBs and IDBs increase with increasing strain, and some of these boundaries may become high-angle boundaries with sufficient strain [21,32–35]. Any mechanism that facilitates the accumulation of stored dislocations will also promote grain refinement [27]. It is also found that during rolling, the texture development and grain orientation also play a role in grain refinement [32,33].

Severe plastic deformation by ECAP introduces the complexity of repetitive passes with varying shear strain planes. Grain refinement under these conditions has promoted the consideration of additional mechanistic details, including the notion of accumulative strain [6] and the effects of variation of shear strain plane [7,9,13]. The latter was further developed from the grain distortion model by Furukawa et al. [16]. These new considerations are helpful and aid the interpretation of certain experimental observations. However, they are not sufficiently general and contradict each other in some cases. Moreover, we believe that texture formation and crystal structure of materials are fundamental elements of ECAP that must be addressed. They are expected to significantly affect the grain refinement as found in cold rolling [32–35]. Strong texture formation has been observed [19,20,36] but has not been systematically studied. We shall focus our following analysis on the interaction of shearing planes with crystal structure and texture formation.

3.1. Shear strain plane

Fig. 2 shows macroscopic shear strain planes for each ECAP route with $\Phi = 90°$ (Fig. 2a) and 120° (Fig. 2b). Note that a similar schematic drawing for $\Phi = 90°$ (Fig. 2a) has been reported by Nemoto et al. [17]. The angles (α) between two adjacent shear strain planes are presented in Table 1. It is interesting to note that both route A from a $\Phi = 120°$ die and route B_C from a $\Phi = 90°$ die have the same α value of 60°. These two combinations of ECAP route and Φ value have been reported to be most effective in refining the grain size of the Al and its alloys. This may make one believe that the 60° is a special α value that most effectively refines the grains of f.c.c. Al. However, this cannot explain why route B_C is more effective than route B_A for a $\Phi = 90°$ die. Although both routes have an α value of 60°, route B_A has no redundant strain and should be more effective than route B_C in grain refinement. Therefore, there is at least one factor other than shear strain plane and accumulative nonredundant strain that significantly affects the grain refinement.

Table 1
The angle (α) between two adjacent ECAP shear strain planes for various ECAP routes

Angle Φ (deg)	α value (deg)			
	Route A	Route B_C	Route B_A	Route C
90	90	60	60	0
120	60	41.4	41.4	0

3.2. Crystal structure

Polycrystalline materials with different crystal structures may respond differently to the same imposed shear strain. Consequently, this will affect the process of grain refinement. The strain plane and value imposed on a material induces responses from specific slip and/or twinning systems in the material. Which slip or twinning system is activated depends both on geometric constraints and crystal structure. For bulk polycrystalline materials, each grain is constrained by its neighbors. Taylor's full constraints model [39] requires five independent deformation systems to maintain material continuity. In general, b.c.c. metals deform by slip since they have many slip systems, which are $\{110\}\langle 111\rangle$, $\{112\}\langle 111\rangle$ and $\{123\}\langle 111\rangle$. F.c.c. metals have fewer primary slip systems, which are $\{111\}\langle 110\rangle$. The deformation mode of f.c.c. metals is also influenced by the stacking fault energy γ_{SFE}. Metals with medium to high γ_{SFE} values such as Cu and Al deform by slip, while those with low γ_{SFE} values such as Ag prefer twinning. Due to the lack of sufficient slip systems, twinning always plays a role in the plastic deformation of h.c.p. metals such as Ti [40–43]. Depending on the c/a ratio of a h.c.p. metal, the slip plane can be the (0001) basal plane (e.g. Be and Zn) or the prismatic $\{10\bar{1}0\}$ planes (e.g. Zr and Ti). The slip direction is primarily $\langle 11\bar{2}0\rangle$.

With limited experimental results [4–9,12–14,23], it is not yet possible to sort out how the shear strain plane affects grain refinement for all materials. However, f.c.c. Al and its alloys have been studied with all four ECAP routes using dies with two Φ angles (90 and 120°). Therefore, we shall focus our analysis on Al and its alloys. The high stacking fault energy of Al makes dislocation slip on $\{111\}$ planes the only deformation mechanism. The $\{111\}$ planes form a tetrahedron, with any two faces intersecting at an angle of 70.5°. The significance of this angle for the grain refinement will be discussed in the following sections.

3.3. Deformation texture

Texture formation during the ECAP processing has not been systematically studied. Heavy texture has been reported in ECAP-processed Al alloy [38], Cu [19,36], Be [37] and Fe [20]. Texture has been found to affect the grain refinement during rolling [31]. Unfortunately, texture has not been systematically studied for any

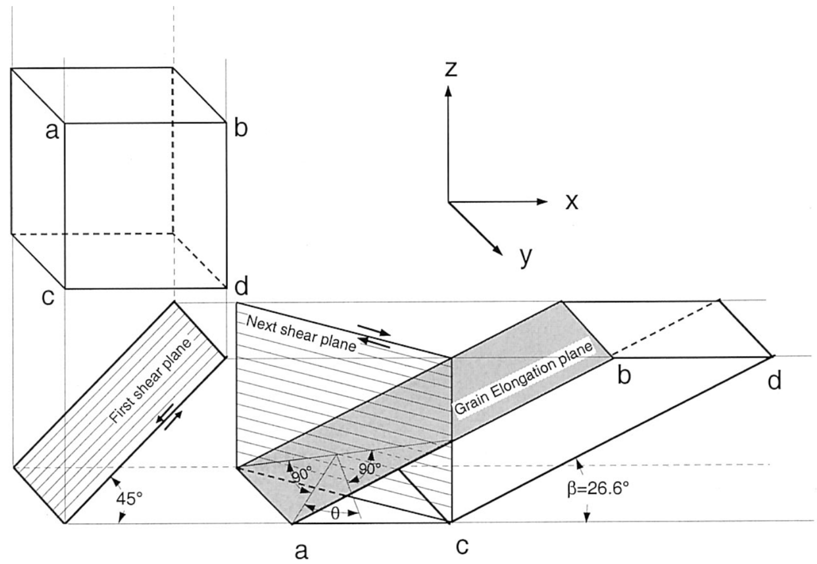

Fig. 3. Orientation relationship between the grain elongation plane of the first pass and the shear plane of the second pass for ECAP route B_C or B_A. The angle between the grain elongation plane and the next shear plane is defined as θ.

metals processed by ECAP. Lacking sufficient texture analysis, we shall discuss the grain elongation effects and their possible relationship with the texture.

Iwahashi et al. [12] developed a model to predict the grain elongation for ECAP routes A, B_C, and C. The model agrees very well with experimentally measured results for Al [12], Ni [14], Cu and Al alloy [4]. Following the same approach, one can also derive the grain elongation direction for route B_A. For a $\Phi = 90°$ die, we have calculated the angles (β) between grain elongation direction and the extrusion axis (x axis) (see Fig. 3) for varying ECAP routes and number of passes. These β values are presented in Table 2.

Stout et al. [44,45] studied the texture formation of copper from torsion, and found that the {111} slip planes tend to become parallel to the shear plane. During ECAP, the shear plane is 45° to the x axis (see Fig. 3). Therefore, one might expect a {111} texture to develop in Al, parallel to the shear plane. However, the die constraint forces a rigid body rotation on the work piece, which makes the {111} texture plane have a smaller angle with the x axis. We can reasonably assume that the {111} texture plane for Al (or slip plane) is oriented between the shear direction and the grain elongation direction. We define the angle between the texture plane and the x axis as ω. After the first pass, the ω can be reasonably assumed to be within a range of 26.6–45°. After more ECAP passes, ω is expected to follow the same trend as β, i.e. ω decreases if β decreases.

Pithan et al. [38] studied Al A5056 alloy, which has maximum pole density at an ω angle of 35° after the first ECA pass. Agnew et al. [19] measured and computationally simulated the {111} pole figures of Cu processed by ECAP. The maximum pole density occurs at $\omega \approx 40°$ after the first pass. These results indicate that the assumption is reasonable for f.c.c. Cu and Al alloy. It also works well with h.c.p. beryllium, whose slip plane is the (0001) basal plane. It has a maximum (0001) pole density at $\omega \approx 30°$ after one ECAP pass, and at $\omega \approx 20°$ after two ECAP passes using route A [37], which are very close to the corresponding β values (26.6 and 14.0°, respectively). These data indicate that the assumption is also reasonable for h.c.p. metals. Note that twinning always plays a role in the deformation of h.c.p. metals. However, the assumption may not work well with b.c.c. metals, which have many slip planes ({110}, {112} and {123}), as indicated by reported results of texture measurements [20].

3.4. Grain refinement mechanisms

Building upon previous discussions of shearing planes, crystal structure, and texture, we are now ready to analyze the grain refinement by various routes and die angles. Since the experimental data are most complete on Al and its alloys, they will be the focus of our

Table 2
Calculated angles (β) between grain elongation direction and the extrusion (x) axis

Number of passes	β value (deg)			
	Route A	Route B_C	Route B_A	Route C
1	26.6	26.6	26.6	26.6
2	14.0	19.5	19.5	Equiaxed
3	9.5	26.6	12.6	26.6
4	7.1	Equiaxed	10.0	Equiaxed

analysis. They are typical of materials with f.c.c. crystal structure and with medium to high stacking fault energy, i.e. dislocation slip on {111} planes is the primary deformation mechanism.

Route B_C for a $\Phi = 90°$ die is reported most effective in refining the grain size of f.c.c. Al and its alloys [7,12,13]. As shown in Table 2, after an odd number of ECAP passes, the grain elongation direction is at an angle of $\beta = 26.6°$ with the x axis. We define the angle between the grain elongation plane and the next shear plane as θ (see Fig. 3). The angle θ can be calculated as 71.6°, which is very close to 70.5°, the angle between two faces of a {111} tetrahedron. If the assumption is correct that ω lies in the range 26.6–45° when $\beta = 26.6°$, the angle between the {111} texture plane and the subsequent shear plane can be calculated to vary from 71.6 to 60°, which is still very close to 70.5°. Therefore, the texture cause a significant fraction of grains to have one of their {111} planes closely oriented to the subsequent shear plane. This makes it easier to activate the slip system on the latter {111} plane to facilitate shear deformation. At the same time, this may promote subgrains to rotate a few degrees, which increases the misorientation of low-angle subgrain boundaries. Therefore, we believe that this unique orientation relationship ($\theta = 71.6°$) between the grain elongation plane and the shear plane promotes effective grain refinement. It can be seen from Table 2 that route B_C retains the $\beta = 26.6°$ value after odd-numbered passes, consequently maintaining the unique orientation relationship between the grain elongation plane and the shear plane. This explains why route B_C is most effective in refining grain size. For simplicity, the θ value is hereafter used to discuss the effectiveness of grain refinement. The closer the θ value to 71.6°, the more effective the grain refinement. Of course, the more appropriate parameter for describing the effectiveness of grain refinement is how close the angle between the texture plane and the shear plane is to 70.5°, the angle between two {111} tetrahedron planes of the f.c.c. Al. However, because of the lack of texture data, the θ value is the best parameter we can use, with the assumption that ω is not deviated too far from β.

The presented analysis makes it easier to explain the behavior of other ECAP routes. Although route B_A yields the same angle α between two adjacent shear planes, the β value continues to decrease from 26.6° after the first pass. Thus, the θ value cannot be maintained at values near the optimum 70.5° for most effective grain refinement. This makes route B_A less effective than route B_C in grain refinement, although route B_A does not have redundant strain.

The θ values for route A ($\Phi = 90°$) can be calculated as 71.6° after the first pass, 59.0° after the second pass, 54.5° after the third pass and 52.1° after the fourth pass. It continues to decrease with increasing number of pass. Therefore, route A is not effective in refining grain size. As discussed later, the effective accumulation of nonredundant strain in route A does not make it more effective than route C in accumulating the stored dislocations.

This analysis has been focusing on ECAP with the die angle $\Phi = 90°$. To explain the behavior of route C, we have to compare route C with route A for both $\Phi = 90$ and 120°. Route C was found more effective in grain refinement than route A for $\Phi = 90°$, but less effective than route A for $\Phi = 120°$. Prangnell et al. [6] used redundant strain argument to explain why route C is least effective in grain refinement for $\Phi = 120°$. For route C, every even-numbered pass completely reverses the shear strains of its previous odd-numbered pass, creating redundant strain. Vatne et al. [26] and Davenport et al. [27] reported that the deformation texture and dislocation structure can, at least partially, be reversed when the shear strain is reversed. This makes route C ineffective in storing the dislocations and refining grain size, which explains why route C is not effective. On the other hand, route A is most effective in grain refinement because it does not generate redundant strain.

The redundant strain theory cannot explain the behavior of routes A and C for $\Phi = 90°$, where route C is more effective in refining grain size than route A. This can only be explained by the assumption that the reversed strain in route C reverses little, if any, dislocation structure or texture. In fact, Rollett et al. [46] strained pure Al by torsion for a shear strain of 2 and subsequently reversed the strain to check the reversibility of texture. They found that the texture is not reversible, which contradicts the results of Vatne et al. [26]. Rollett and Wright [40] argued that the dislocation motion through the dislocation 'forest', and the accumulation of stored dislocation is not reversible. Coincidentally, the shear strain from a $\Phi = 90°$ ECAP die can be calculated as 2 using Eq. (1), the same as the shear strain used by Rollett and Wright [40]. Therefore, we expect the accumulation of stored dislocations and the texture to be irreversible for Al processed using a $\Phi = 90°$ ECAP die. On the other hand, for a $\Phi = 120°$ ECAP die, the shear strain is only 1.15. One may reasonably expect that it is easier to reverse the accumulation of stored dislocation and the texture generated by a smaller shear strain, which explains the observation of Prangnell et al. [6].

Routes B_A and B_C are less effective than route A in refining grain size for $\Phi = 120°$. This could be because the angle between the grain elongation plane from the first pass and the second shear plane is 62.4°, which is not as close to the unique $\theta = 70.5°$ value. Interestingly, route B_C is slightly more effective than route

B_A, although route B_C has redundant strain. Davenport et al. [27] argued that the rotation of billet in route B_C causes cross-hardening by the interactions of mobile dislocation with geometrically necessary dislocation. These interactions make it difficult to reverse the dislocation accumulation, which explains the effectiveness of route B_C in grain refinement despite its redundant strain.

4. Issues and suggested research

In the presented analysis of the shear plane effect on the grain refinement, an assumption has been made that the deformation texture orients the slip plane between the shear direction and the grain elongation direction. Such an assumption agrees well with f.c.c. metals such as Al alloy and Cu, as well as h.c.p. metals such as Be, but may not agree with results for b.c.c. metals. Another issue is the reversibility of dislocation accumulation and texture during the ECAP process with increasing shear strain from a single pass. This can be studied by varying the die angle Φ. For example, the shearing strain for a single pass varies from 0.4 to 2 when die angle is varied from 157.5 to 90° in the work of Nakashima et al. [9].

Texture evolution associated with various ECAP routes has so far not received much attention. Because of its primary role in microstructural development and, more specifically, grain refinement, the texture evolution needs to be focused on in future research. We recommend the study of the texture evolution of the following materials using all four ECAP routes: (a) f.c.c. metals with medium to high stacking fault energy, such as Al and Cu; (b) f.c.c. metals with low stacking fault energy, such as brass (70Cu:30Zn); (c) b.c.c. metals such as Fe; (d) h.c.p. metals with a prismatic slip plane such as Ti; and (e) h.c.p. metals with a basal slip plane such as Zn.

5. Summary

The grain refinement during the ECAP process is affected by accumulative strain and the interaction of shearing plane with crystal structure and deformation texture. For f.c.c. metals with dislocation slip as their primary deformation mechanism, the latter plays a dominant role with a $\Phi = 90°$ die, and the former plays a dominant role with a $\Phi = 120°$ die. We believe that texture evolution and its relationship with the shear plane are key factors that affect the grain refinement. Finally, we have suggested further investigations on the texture and grain refinement of metals with various crystal structures and deformation mechanisms.

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